"Fabrication and Characterization of Metal/Metal Oxide-Graphene Oxide/reduced Graphene Oxide Nanocomposites"

A Synopsis Submitted to

Research Advisory Committee

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Synopsis

Fabrication and Characterization of Metal/Metal Oxide-Graphene Oxide/reduced Graphene Oxide Nanocomposites

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INTRODUCTION

Graphene is a two-dimensional zero bandgap nanomaterial derived from three-dimensional graphite. It owns the hexagonal honeycomb lattice structure composed of a monolayer of carbon atoms and has a bond length of 0.142 nanometres. Graphene has become the most trending nanomaterial in the field of research as it shows remarkable optoelectronic, mechanical, thermal, physical, and chemical properties. In addition, it is the lightest, strongest, and thinnest material. It is the best conductor of heat and electricity. Graphene oxide (GO) is an oxidized derivative of graphite material and has layers of graphite holding numerous oxygen containing functional groups. After the removal of these functional groups, obtained reduced graphene oxide behaves equivalent to the graphene.

The concept of "Composite" is existing for long. It simply means the combination of two or more materials having different chemical and physical properties. This combination aims to get material with more advanced properties than before. Later, nanocomposites were introduced to fulfill most of the social needs in the field of science. Nanocomposites differ from ordinary composites because it has at least one nano range phase and has a high surface to volume ratio. Within no time nanocomposites become popular among researchers, being a multifunctional, easily controllable, best performer and having unique physical and chemical properties. Among various nanocomposites, Graphene based Metal/Metal Oxide Nanocomposites are attracting many scientists as they are behaving exceptionally good in comparison with the rest of the materials.

The name 'metal oxide' itself tells that it comprises of the oxide anion and metal cation. They are commonly known as basic oxides and exist in the form of crystalline solids. In the past few years, metal oxides are widely been studied being a promising candidate for a variety of applications such as energy storage devices, sensors, catalysis, etc. Some examples of metal oxides are CuO [1,2], ZnO [3], Fe_2O_3 [4], SnO_2 [5,6], ZrO_2 [7,8] and TiO_2 [4,9] used for nanoparticle synthesis. Among them, Zinc Oxide (ZnO) is a rapidly rising wide energy bandgap semiconductor material of group II-IV in a periodic table

for a wide range of applications such as diluted magnetic semiconductors [10], light emitting diodes [10], photocatalysts, etc.

Also, the researchers are engaged in studying the Graphene based metal NCs seeing their significant contribution in various applications like the field of water treatment [11], electrochemical energy storage applications [12], environmental applications [13], etc.

REVIEW OF LITERATURE

The ‘graphene’ word originated from the Greek word ‘graphe-in’ which means ‘to write’. In the year 2004, two scientists Konstantin Novoselov and Andre Geim discovered this outstanding material graphene through the Scotch tape method and received the Nobel Prize in physics for this discovery in the year 2010. The term 'graphene' is simply a single flat layer of carbon atoms densely arranged in a 2D lattice of honeycomb shape [14]. It serves as a building block for graphite carbon materials. For example, 0D fullerene i.e., cage structure, 1D nanotubes i.e., rolled graphene sheets, and 3D graphite i.e., stacked graphene sheets. When every single layer of graphite is treated as an independent structure, it shows unique physical, chemical, thermal, mechanical, and electrical properties [15]. Also, it has 5000 $Wm^{-1}K^{-1}$ high thermal conductivity, 2630 m^2g^{-1} large surface area, 1 TPa high young modulus, 250,000 $cm^2 V^{-1}s^{-1}$ high electron mobility, better optical transmittance, and electrical conductivity [16-20]. These exceptional properties of graphene open a new road for various applications such as electrochemical sensing, solar cells, OLED displays, data storage, etc [21].

To date, there is a lot of work done to merge graphene with the various metal oxides and metals. Among them, the Graphene based ZnO NCs and Metal-graphene NCs are being focused nowadays. It is said that zinc oxide engulfed in Graphene oxide prohibits the formation of graphene clusters and graphene controls the dispersion and stability of zinc oxide nanoparticles [22]. Therefore, graphene oxide and zinc oxide together can make fascinating nanocomposites. During the synthesis process of NCs, the reduced graphene oxide (rGO) is used as a graphene alternative. The only reason is its low cost and large-scale production [23]. The preparation techniques used for ZnO-GO nanocomposites are the co-precipitation method [24], the solvothermal method [25], the microwave-assisted method [26], the chemical precipitation method and

environmental dye remediation [27], one step photochemical method [28], and has applications in antibacterial agent development [24], optoelectronic devices [29].

When graphene is merged with inorganic nanomaterials such as ZnO, cobalt, Al_2O_3, iron oxide, and Nikel, it transmits electronic properties along with magnetic properties in the final structure of 3D printing [30]. This makes the printed structures useful in the anodes of Li batteries, biosensors, electrochemical double-layer supercapacitors, and many more. The graphene-assisted graphitic ZnO layers showed that single-layer ZnO jammed structures have less steadiness than multilayer packed structures [31]. It is predicted that the inexpensive advanced temperature sensors, developed by merging graphene with smart temperature sensors, will be the best replacement for the traditional sensors [32]. The photocatalytic efficiency of Graphene ZnO nanocomposites was found to be one step ahead of the ZnO nanoparticles [27]. Yao et al. found that the addition of G-ZnO in the high-density polyethylene (HDP) influenced the crystallinity of HDP and gains high thermal stability, best barrier performance, better antibacterial capacity and mechanical properties which is helpful in the field of packaging [33]. The nanofluids based on the rGO-ZnO NCs can be used in the heat transfer application [34].

Metal-graphene NCs are being studied widely due to their excellent performance. It is said that graphene is a perfect choice for the union with metal nanostructures. This is because graphene has outstanding intrinsic properties like large surface area, strong optical absorption, and adsorption of molecules [35,36]. Noble metal, silver (Ag), is the most trending metal for scientific research. The reason behind this is that the combination of Ag with graphene exhibits catalytic, optical, and antibacterial properties having a wide range of applications in catalysis, sensors, and environmental treatments respectively [37-39]. One more noble metal i.e., Gold (Au) has extraordinary conductivity and great catalytic properties. That is why Au and Ag become the most promising metal to work on metal-graphene NCs. The preparation methods for metal-graphene NCs are the hydrothermal method [13,40], green synthesis approach [11,12], simple room temperature stirring method [41], rapid thermal reduction method [42], etc.

Kwon *et al.* uses the 3D printing process for the fabrication of multiaxial piezoresistive sensors to detect human motion. For this, they used graphene nanoplatelets, silver nanoparticles, and polyurethane [43]. Weiwei *et al.* synthesized Ag-graphene NCs and

how the morphology and microstructure were affected because of the Ag content are studied [44]. Wang *et al.* have synthesized the Ag-graphene NCs via a one-step irradiation strategy to use as a lubricant additive to reduce friction and wear [45].

During the research, the researcher Wang *et al.* synthesized the Au graphene nanocomposites via the UV irradiation synthesis technique. They investigated morphological, optical, and electrochemical properties for their electrochemical sensing applications [46]. analysis of Bisphenol A is done by Gugoasa *et al.* with the help of a gold-reduced graphene oxide composite paste electrode fabricated by a 3D printing process [47]. Gold-graphene oxide NC is synthesized in a supercritical carbon dioxide fluid by Meng *et al.* They discussed structural, optical, and thermal gravimetric analysis on prepared oxide NCs and proposed it as a future energy-efficient lubricant additive [48].

Nowadays, the 3D printing film deposition method [49] is a newly accepted method to produce graphene-based metal/metal oxide nanocomposites. Design flexibility is the advantage of this method. The complex 3D nanostructures can be constructed with a very small amount of material, minimum processing time, and provides greater improved qualities.

RATIONALE

In graphene, carbon atoms are Sp^2 hybridized. Sp^2 hybridization of the atoms makes the conduction of electrons easier. This electronic conduction in graphene can be upgraded by engulfing various metal and metal oxide nanoparticles. Also, the interdependent properties between graphene and metal/metal oxide nanoparticles can be improved. This improved electronic conduction and the interdependent properties serve great potential in the range of applications.

According to the review of the literature, the 3D printing process is not yet popular for thin film deposition. Also, the synthesis of ZnO-GO NCs in powder form is done by many scientists. So, during this PhD work, the 3D printing process will be used for preparing the NCs and the ZnO-GO NC thin film will be prepared and studied.

OBJECTIVE

The primary goal of this thesis is to fabricate Graphene Oxide (GO) and its metal and metal oxide nanocomposites thin films using the 3D-printing technique. The precursor GO will be synthesized by modified Hummer's method and spot reduction of GO will be done by electrochemical method. The solutions of metal (Au and Ag) salts will be directly used as a precursor for metal-GO nanocomposites. On the other hand, for metal oxide (ZnO) GO nanocomposite, the metal oxide will be prepared by chemical route using the co-precipitation method and then solution of the prepared metal oxide will be incorporated in the GO solution to make GO nanocomposites.

Thus, the objective of the thesis can be as:

1. Synthesis of GO by modified Hummer's method and spot reduction of GO via the electrochemical method.
2. Preparation of metal oxide nanomaterials by chemical route.
3. Deposition of Ag-GO/rGO nanocomposite with different molar concentrations (1, 2, 3, 5, 10) of Ag with respect to GO.
4. Deposition of Au-GO/rGO nanocomposite with different molar concentrations (1, 2, 3, 5, 10) of Au with respect to GO.
5. Deposition of ZnO-GO/rGO nanocomposite with different molar concentrations (1, 2, 3, 5, 10) of ZnO with respect to GO.
6. All the metals and ZnO will be also deposited with different molar concentrations without the GO matrix for comparison.
7. To study the effect of Ag, Au, and ZnO content in their respective Go nanocomposites on the morphological, structural, optical properties and their electrochemical performance.
8. Fabrication of metal and metal oxide-GO/rGO nanocomposite thin films at different applied voltages.
9. To study the effect of the applied voltages on the morphological, structural, and optical properties of fabricated nanocomposite thin films.
10. For the application point of view, the prepared metal/metal oxide-GO/rGO nanocomposite will be used to manufacture an electrode for the supercapacitor.

3D printing film deposition requires a conductive substrate. Hence, the Indium Tin Oxide coated glass will be used as a substrate for all the sample preparation.

To achieve the above-mentioned goals, different characterization techniques such as scanning electron microscopy, x-ray diffraction, diffuse reflectance spectroscopy, Raman spectroscopy, x-ray absorption spectroscopy, cyclic voltammetry and Extended x-ray absorption fine structure spectroscopy will be used.

RESEARCH METHODOLOGY

Synthesis techniques

Improved Hummer's Method

Graphene oxide (GO) will be synthesized via improved Hummer's method [50]. This is the well-established chemical method to prepare GO using potassium permanganate, H_2SO_4, H_3PO_4, and graphite powder.

3D printing film deposition

3D printing technology i.e., Additive Manufacturing (AM) is the research area where 3D objects are constructed through layers addition. In the last two decades, nanoparticles are engulfed into a polymer matrix leading to electrical properties, higher thermal stability, great mechanical stability, higher specific surface area. 3D printed materials provide a wide range of applications in membranes, conductive materials, photochromatic materials, etc. It consists of a nozzle which is filled with electrolyte solution comprised of metal ions, and a conductive substrate, which is biased related to a quasi-reference counter electrode in the capillary. The nozzle is placed accurately with the help of an integrated micro and nano positioning system.

Co-precipitation method

The coprecipitation method is an inexpensive, simple synthesis technique for nanoparticles. According to the requirement, the solution of inorganic metal salt and water will be prepared. As a result, metal cations take the form of metal hydrate species. pH of the solution is then lowered aiming at the condensation of these hydrates. After performing the processes like filtration, washing, and drying sequentially, the nanoparticles are formed.

Characterization techniques

X-ray Diffraction

XRD is the most important non-destructive characterization technique widely used in science to study both amorphous and crystalline materials on an atomic scale. It helps to find out the crystal structure of the material and the crystallite size, chemical composition, lattice strain as well as the state order. This method includes the X-ray irradiation of the sample followed by the measurement of scattering angles and intensities of the scattered X-rays. The material structure is determined from the intensity versus scattering angle plot. The Rietveld refinement method is used not only to get information like Miller indices, angles, intensities but also to know how exactly the obtained structure deviates from the standard one. In this work, XRD will confirm the structure of prepared nanocomposites.

Scanning electron microscopy

Observation of surface morphology such as particle size, microstructure, particle shape and external morphology can be done through the technique called scanning electron microscopy (SEM). In this characterization technique, the prepared sample is scanned with the help of an ultra-fine beam of electrons in a parallel lane. Thus, the interaction of the electrons with the sample gives rise to a variety of signals and X-rays. These signals are then detected and form the digitized image of the sample.

Diffuse reflectance spectroscopy

Diffuse reflectance spectroscopy is the easier, faster, and more systematic type of absorption spectroscopy for the analysis of a variety of samples such as crystalline materials or troublesome solid samples. The opaque samples having strong absorbing ability are examined via this technique. In this method, light reflected from the sample is measured rather than the light transmitted from the sample. The incident light beam on the sample surfaces undergoes reflection of two types i.e., specular reflection and diffuse reflection. Among these reflections, the DRS technique studies the diffusely reflected beam.

The obtained reflectance spectrum from the DRS method is very helpful to get the idea about the optical bandgap (E_g) of the material. The Kubelka-Munk plot is the graph

between the absorption coefficient vs photon energy (hν). Thus, extrapolation of the linear region to the horizontal axis yields the energy (E_g) of the material.

Cyclic voltammetry

Cyclic voltammetry (CV) is an analytic technique used to study how the prepared material has electrochemical performance. It comprises of the electrode potential ramped linearly versus time forming a triangular waveform and cyclic voltammogram created by recording the current as a function of potential. Three electrodes connected to the potentiostat were immersed in an analyte-electrolyte solution. This whole assembly is said to be an electrochemical cell and is used to perform voltametric measurements. The three electrodes are the working electrode on the surface on which redox reaction occurs, the counter electrode conducting the electricity and last is the reference electrode that gives the fixed potential for the measurement of the working electrode potential. The study of the electronic properties of the material can be done by measuring the redox potential. Factors like the shape of the voltammogram, peak current, and peak potential serve the electrochemical properties of the redox system.

X-ray absorption spectroscopy and Extended x-ray absorption fine structure

X-ray absorption spectroscopy abbreviated as XAS is a famous technique used for the determination of the electronic structure of the matter or local geometry. This technique requires intense x-ray beams so it can be performed with synchrotron radiations. It measures the transitions from metal's electronic states present in the core to the excited electronic states. This technique is classified as XANES (X-ray absorption near-edge structure) and EXAFS (Extended x-ray absorption fine structure). EXAFS gives information about the types, numbers, and distances to ligands as well as the nearest atoms from the absorbing elements. The metal site symmetry and electronic structure can be determined through XANES.

Raman spectroscopy

Raman spectroscopy provides both chemical and structural information, as well as takes part in the identification of substances through their corresponding characteristic Raman fingerprint. Raman spectroscopy finds out this information via the detection of Raman scattering from the sample. When the incident light is scattered by a molecule, the molecular electron cloud is polarized by the oscillating electromagnetic field of a photon. As a result, the molecule is in a higher energy state by the transfer of photon energy to the molecule. Every peak in Raman spectra corresponds to the vibrational energy of bonds in a carbon compound. In the case of graphene, the Raman spectrum shows three typical peaks mainly D peak, G peak and 2D peak at 1350 cm^{-1}, 1580 cm^{-1} and 2690 cm^{-1} respectively. But the D peak never be there in the pure graphene spectrum. While, the Raman spectrum of graphene oxide shows only two peaks i.e., D peak at 1353cm^{-1} and G peak at 1605cm^{-1}.

Therefore, even a small structural morphology can be identified through the Raman spectra. Since all carbon nanomaterials have the same structure, Raman spectroscopy helps us to distinguish them from one another.

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